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QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR--ETC(U)
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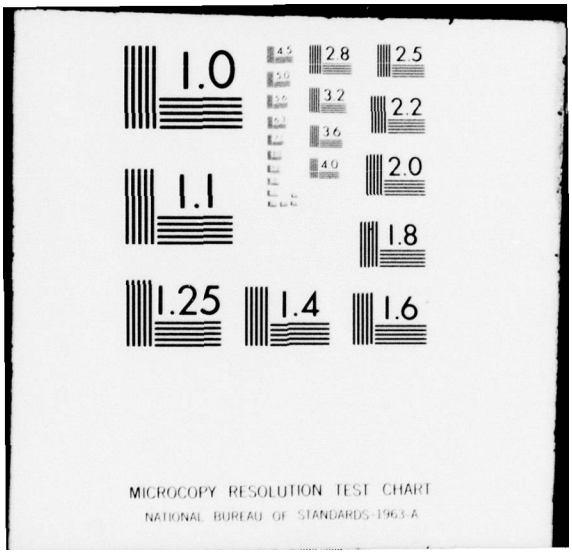
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# QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR MINUTEMAN STAGE 1 UF-2121 LINER

PROPELLANT LABORATORY SECTION



MANCP REPORT

NR 401 (78)

AUGUST 1978

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(6) QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR MINUTEMAN STAGE 1 UF-2121 LINER

(9) Semi-annual rept.

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## ABSTRACT

Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

This report includes the test results for the first, second, third and fourth time testing of the control and special specimens at Ogden ALC.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 REV A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

TABLE OF CONTENTS

# INTRODUCTION

A. PURPOSE:

Quality assurance testing of specimens prepared from the new source of MAPO to assure that liner material for First Stage Minuteman Motors will perform as predicted.

B. BACKGROUND:

*Tris [1-(2 methyl) aziridinyl] phosphine oxide (MAPO) is used as a curing agent in the Minuteman Stage One UF-2121 liner. MAPO was produced by Immont Chemical (Immont) and shipped to Arsynco Incorporated (Arsynco) for purification and marketing. Immont sold the production rights for MAPO to Arsynco and terminated the production of raw MAPO in 1972.

Since MAPO is a critical ingredient in UF-2121 liner formulation, the source change for the manufacture of MAPO was considered a serious change. Therefore, it was necessary for Thiokol to conduct qualification testing on liner material using MAPO manufactured by Arsynco before it could be considered acceptable for use in Minuteman Stage I UF-2121 liner.

ERLA-500 was the qualified epoxy curing agent used with MAPO in the UF2121 liner. Union carbide terminated their process for ERLA-500. ERLA-510 used in similar liners (i.e. UF-2137) was substituted for ERLA-500 and qualified with MAPO from the new source.

The test conditions and test methods are shown in Table I.

*TWR-7857 Rev A Report, J. W. Rabern

Qualification testing was performed by Thiokol and reported in TWR-7857 Rev A. In addition, specimens were prepared by Thiokol from the new vendor's material and from the old source material for a "follow on" test program. These specimens were then transferred to Ogden ALC for a continuing surveillance test program designed to cover a ten year span. The material from the old source will be used as the control samples.

The ten year sampling plan is shown below. Those specimens identified for the fourth year were tested at this test period. The types of specimens are Disc (steel/liner/steel), Cup (steel/liner/TP-H1011), and Peel (broadcloth/liner/TP-H1011). For the disc specimen, the adhesion between the liner and steel is the critical factor. For the cup specimen, the adhesion between the propellant and the liner is critical. For the peel specimen the propellant to liner peel strength when pulled at 180° is critical.

Storage

TEN YEAR CONTINGENCY AGING SAMPLE CODING

| Age (yr) | Temp (°F) | Disc (Sample Nr) | | Cup (Sample Nr) | | Peel (Sample Nr) | |
|---|---|---|---|---|---|---|---|
| | | Control* | Special** | Control* | Special** | Control* | Special** |
| 1 | 75 | 1- 6 | 181-186 | 61- 66 | 241-246 | 121-126 | 301-306 |
| 2 | 75 | 7-12 | 187-192 | 67- 72 | 247-252 | 127-132 | 307-312 |
| 3 | 75 | 13-18 | 193-198 | 73- 78 | 253-258 | 133-138 | 313-318 |
| 4 | 75 | 19-24 | 199-204 | 79- 84 | 259-264 | 239-144 | 319-324 |
| 5 | 75 | 25-30 | 205-210 | 85- 90 | 265-270 | 145-150 | 325-330 |
| 6 | 75 | 31-36 | 211-216 | 91- 96 | 271-276 | 151-156 | 331-336 |
| 7 | 75 | 37-42 | 217-222 | 97-102 | 277-282 | 157-162 | 337-342 |
| 8 | 75 | 43-48 | 223-228 | 103-108 | 283-288 | 163-168 | 343-348 |
| 9 | 75 | 49-54 | 229-234 | 109-114 | 289-294 | 169-174 | 349-354 |
| 10 | 75 | 55-60 | 235-240 | 115-120 | 295-300 | 175-180 | 355-360 |

* Liner mix A73-11846 - control or old MAPO source material

** Liner mix A73-11810 - Experimental MAPO

TABLE I

Test Conditions and Methods

| Group | Test | Condition | Config-uration | G085 Spec Code | Spec Per Cond | Total Number of Spec | Test Method |
|---|---|---|---|---|---|---|---|
| Bond in Tension Disc | Tensile Adhesion OI#127-3 | CHS 0.5 in/ min, Chart 5.0 in/min, 500 lbs full scale load 77°F ± 2° | Discs | TV | Control 6 Special 6 | 12 | A |
| Bond in Tension Cup | Tensile Adhesion OI#127-3 | CHS 0.5 in/ min, Chart 5.0 in/min, 200 lbs full scale load 77°F ± 2° | Cup | TC | Control 6 Special 6 | 12 | A |
| 180° Peel Specimens | Tensile Peel OI#127-3 | CHS 10 in/min 77°F ± 2° Chart 5 in/ min 20 lbs full scale load | Peel | TE | Control 6 Special 6 | 12 | B |

TEST CONDITIONS

A. Testing of tensile adhesion specimens was performed using an Instron testing instrument. Properties measured were maximum stress to the nearest pound and failure mode.

Steel disc specimens require a stress of about 240 psi. The recommended initial full scale load is 500 pounds. This instrument setting should be changed to another setting if the first reading goes off scale on the high side. If 500 psi is exceeded, then change the reading to 1000 psi full scale.

Cup adhesion specimens are tested with a stress of 200 lbs per sq inch; the recommended full scale load is 500 lbs.

B. Testing of 180° peel samples was performed using an Instron testing instrument. The physical property of the material to be determined was the average peel strength to the nearest pound per inch.

NOTE: Thiokol's procedure for Testing and Laboratory Mixing of UF-2121 Liner. SLP 400, 28 April 71.

## STATISTICAL ANALYSIS

UF-2121 liner material is being tested under a ten year program to determine whether or not differences exist between liner materials manufactured from two separate sources of curing agent (MAPO). Test specimens were manufactured in two groups; control, using original source curing agent, and special, using new source curing agent. The test specimens for these two groups are of three kinds; disc, cup, and peel. For each specimen type within each test group the sample test size is six. Laboratory testing for four test periods or four years has been accomplished. Test data for the years 1975, 1976, 1977, and 1978 are in Tables 2, 3, 4, and 5 and data columns are summarized using means and standard deviations.

To determine any differences in data pertaining to the two MAPO sources, statistical analysis should take into consideration the effect of differing test periods or aging. Two factor analysis of variance was employed to analyze effects pertaining to MAPO source and test period, see Tables 6, 7, and 8. Hypotheses were made that the MAPO source has no effect on the data and also that the test period has no effect. If a hypothesis fails, then it is termed significant. Analysis of variance indicates significant test period differences in data pertaining to each of the three specimen types while the MAPO source is only significant in the cup data, see Table 9. A significant statement for interaction is given when the trend from control to special data in one test period is opposite the trend found in another test period.

With four test periods accomplished, regression plots (Figures 1 - 6) were made to determine whether slope differences existed between control and special test data. No differences between slopes were found. The regression model $Y = a + bx$, using individual data points, was employed to evaluate test results. The variance about the least squares trend line is used to compute a tolerance interval such that at the 90% confidence level 90% of the sample distribution falls within this interval. This tolerance interval is extrapolated 24 months past the age point pertaining to the oldest motor tested. The statistical significance of the slope of the trend line is evaluated for each regression plot. If significant, it is an indication that a change over time is occurring.

## TEST RESULTS

The 1978 test data and the mean for the respective control and special data is shown in Table 5. In addition, for a convenient comparison, the 1975, 1976, and 1977 test data are included in Tables 2, 3, and 4 respectively.

A statistical analysis was made on the 1978 data and the two factor analyses of variance are shown in Tables 6, 7, and 8. Significance at the five percent significance level pertaining to analysis of variance tables is shown in Table 9.

DISC:

No significant difference is shown for variance in MAPO source with a statistically significant difference shown for test period and interaction analysis (Table 9). For the year 1978, the mean of the control and special data is 14.78 and 14.50 kg/sq cm respectively.

The minimum specification requirement according to TWR-7857 Rev A is 12.30 kg/sq cm minimum. As seen in Table 5, MANCP's data is well above the minimum.

Regression curves for the control and special specimen data shows a statistically significant decrease (Figures 1 and 2). The slopes of the respective curves show no statistically significant difference.

The failure mode for the control and special specimens was 100% cohesive in the liner, except for one special specimen, which was 98% cohesive in liner and 2% adhesive liner to disc.

CUP:

There is a statistically significant difference in MAPO source variance analysis and test period analysis and no statistical significance for

interaction analysis (Table 9).

According to TWR-7857 Rev A Report, the minimum requirement is 4.92 kg/sq cm. The 1978 data means are 10.58 kg/sq cm for the control and 10.41 kg/sq cm for the special specimens.

Regression curves for the control and special specimen data shows a statistically significant decrease (Figures 3 and 4). The slopes of the respective curves show no statistically significant difference.

The failure mode for specimens tested in 1978, with respect to cohesive failure in the propellant and adhesive propellant to liner failure, shows a considerable difference in the respective percentage of failure compared to 1977 data. The 1978 data are shown in Table 10.

The failure mode for the control specimens for 1977 was 100% cohesive in the propellant. The special specimens were 100% cohesive in the propellant for four specimens, 75% cohesive in the propellant and 25% adhesive propellant to liner for one specimen, and for the remaining specimen 90% cohesive in the propellant with 10% adhesive propellant to liner.

PEEL:

There is no significant difference in MAPO source and A and B interaction data between the control and special specimen data. A statistically significant difference is shown for test period (Table 9).

Thiokol reported (TWR-7857 Rev A) 0.679 and 0.732 *kg/L cm respectively for the control and special specimen mean data at age six months. This compares with 1978 data of 0.976 and 1.015 respectively for control and special mean data.

The regression curves for the control and special data show a statistically

significant increase (Figures 5 and 6). The slopes of the respective curves show no statistically significant difference.

The mode of failure was 100% liner to propellant bond.

* Kilograms per linear centimeter

## CONCLUSIONS

Based on this anlaysis, there is no significant difference between MAPO sources pertaining to data for disc and peel specimens. There is a statistically significant difference between the control and special MAPO source test data for cup specimens. This may be caused by an increase in the cohesive failure of the propellant as seen in the failure mode.

For the test period analysis, a statistically significant difference is shown in the test data for disc, cup and peel specimens.

The interaction analyses show a statistically significant difference for disc data with no significant difference for the cup and peel specimen data.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 Rev A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

## RECOMMENDATIONS

It is recommended that the testing plan be continued to assure long range capability of the liner produced from new source material.

TABLE 2

TEST DATA SUMMARY
JULY 1975

| DISC | | | | CUP | | | | PEEL | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | | Special | | Control | | Special | | Control | | Special | |
| Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | Kg/L-Cm | Nr | Kg/L-Cm |
| 1 | 17.085 | 181 | 17.225 | 61 | | 241 | 11.601 | 121 | 0.6786 | 301 | 0.6786 |
| 2 | 17.507 | 182 | 17.858 | 62 | 11.812 | 242 | 11.601 | 122 | 0.6786 | 302 | 0.6965 |
| 3 | 17.225 | 183 | 17.015 | 63 | 11.741 | 243 | 11.531 | 123 | 0.7143 | 303 | 0.6965 |
| 4 | 17.929 | 184 | 16.944 | 64 | 12.163 | 244 | 11.671 | 124 | 0.7500 | 304 | 0.6965 |
| 5 | 17.366 | 185 | 17.436 | 65 | 12.234 | 245 | 11.390 | 125 | 0.7679 | 305 | 0.7143 |
| 6 | 17.296 | 186 | 19.054 | 66 | 11.882 | 246 | 11.390 | 126 | 0.7858 | 306 | 0.6965 |
| $\bar{Y}$ | 17.401 | | 17.589 | | 11.966 | | 11.531 | | 0.7292 | | 0.6965 |
| S | 0.2943 | | 0.7899 | | 0.2191 | | 0.1176 | | 0.0458 | | 0.0113 |

TABLE 3

TEST DATA SUMMARY
MAY 1976

| DISC | | | | CUP | | | | PEEL | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | | Special | | Control | | Special | | Control | | Special | |
| Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | Kg/L-Cm | Nr | Kg/L-Cm |
| 7 | 15.116 | 187 | 14.483 | 67 | | 247 | 13.288 | 127 | 0.9643 | 307 | 1.0536 |
| 8 | 16.311 | 188 | 14.905 | 68 | 13.710 | 248 | 13.710 | 128 | 0.9286 | 308 | 1.0358 |
| 9 | 15.397 | 189 | 14.483 | 69 | 13.640 | 249 | 13.640 | 129 | 0.9286 | 309 | 1.0179 |
| 10 | 15.960 | 190 | 14.765 | 70 | 13.007 | 250 | 13.077 | 130 | 1.0179 | 310 | 1.0358 |
| 11 | 15.819 | 191 | 15.468 | 71 | 13.148 | 251 | 13.359 | 131 | 1.1072 | 311 | 1.0536 |
| 12 | 14.554 | 192 | 14.765 | 72 | 13.499 | 252 | 13.499 | 132 | 1.0001 | 312 | 1.0358 |
| $\bar{Y}$ | 15.526 | | 14.812 | | 13.401 | | 13.429 | | 0.9911 | | 1.0388 |
| S | 0.6356 | | 0.3633 | | 0.3088 | | 0.2354 | | 0.0675 | | 0.0134 |

NOTE: Kg/L-Cm = Kilograms per linear centimeter. Also, for the peel test the results are in average peel.

TABLE 4

TEST DATA SUMMARY
APRIL 1977

| | DISC | | | | CUP | | | | PEEL | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Control | | Special | | Control | | Special | | Control | | Special | |
| Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | Kg/L-Cm | Nr | Kg/L-Cm |
| 13 | 17.155 | 193 | 17.436 | 73 | 9.281 | 253 | 9.140 | 133 | 0.7322 | 313 | 0.7858 |
| 14 | 16.522 | 194 | 16.874 | 74 | 9.281 | 254 | 9.070 | 134 | 0.7143 | 314 | 0.7858 |
| 15 | 16.874 | 195 | 16.944 | 75 | 8.999 | 255 | 8.999 | 135 | 0.6786 | 315 | 0.7858 |
| 16 | 17.015 | 196 | 17.366 | 76 | 9.281 | 256 | 9.140 | 136 | 0.7500 | 316 | 0.7143 |
| 17 | 16.874 | 197 | 17.015 | 77 | 9.492 | 257 | 8.367 | 137 | 0.7500 | 317 | 0.7500 |
| 18 | 16.874 | 198 | 17.015 | 78 | 9.281 | 258 | 8.789 | 138 | 0.6429 | 318 | 0.7143 |
| $\bar{X}$ | 16.886 | | 17.108 | | 9.269 | | 8.918 | | 0.7113 | | 0.7560 |
| S | 0.2107 | | 0.2337 | | 0.1570 | | 0.2994 | | 0.0429 | | 0.0352 |

TABLE 5

TEST DATA SUMMARY
JUNE 1978

| | DISC | | | | CUP | | | | PEEL | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Control | | Special | | Control | | Special | | Control | | Special | |
| Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | $Kg/Cm^2$ | Nr | Kg/L-Cm | Nr | Kg/L-Cm |
| 19 | 14.906 | 199 | 14.554 | 79 | 10.898 | 259 | 10.406 | 139 | 0.9109 | 319 | 1.0180 |
| 20 | 14.624 | 200 | 14.695 | 80 | 10.968 | 260 | 10.476 | 140 | 0.9823 | 320 | 1.0359 |
| 21 | 14.695 | 201 | 14.343 | 81 | 10.617 | 261 | 10.616 | 141 | 0.9466 | 321 | 1.0180 |
| 22 | 14.906 | 202 | 14.343 | 82 | 10.125 | 262 | 10.125 | 142 | 1.0002 | 322 | 1.0359 |
| 23 | 14.343 | 203 | 14.624 | 83 | 10.406 | 263 | 10.687 | 143 | 1.0716 | 324 | 1.0537 |
| 24 | 15.187 | 204 | 14.414 | 84 | 10.476 | 264 | 10.125 | 144 | 0.9466 | 326 | 0.9287 |
| $\bar{X}$ | 14.777 | | 14.496 | | 10.582 | | 10.406 | | 0.9764 | | 1.0150 |
| S | 0.2898 | | 0.1503 | | 0.3166 | | 0.2391 | | 0.0561 | | 0.0444 |

TABLE 6

TWO FACTOR ANALYSIS OF VARIANCE
FOR
DISC DATA, FACTOR A = MAPG SOURCE (CONTROL AND SPECIAL), FACTOR B = TEST PERIOD

| SOURCE | DF | SS | MS | F |
|---|---|---|---|---|
| FACTOR A | 1 | 0.2531713[illegible]E+00 | 0.25917132E+00 | 0.13945827E+01 |
| FACTOR B | 3 | 0.6903[illegible]147E+02 | 0.23003049E+02 | 0.1267[illegible]127E+03 |
| A X B INTERACTION | 3 | 0.177[illegible]324E+01 | 0.59207747E+00 | 0.3261[illegible]4317E+01 |
| ERROR | 40 | 0.72[illegible]E+01 | 0.1815[illegible]908E+00 | |
| TOTAL | 47 | 0.785[illegible]114E+02 | | |

TABLE 7

TWO FACTOR ANALYSIS OF VARIANCE
FOR
CUP DATA, FACTOR A = MAPO SOURCE (CONTROL AND SPECIAL), FACTOR B = TEST PERIOD

| SOURCE | DF | SS | MS | F |
|---|---|---|---|---|
| FACTOR A | 1 | 0.5569330?E+00 | 0.5569330?E+00 | 0.82905960E+01 |
| FACTOR B | 3 | 0.1024?312E+03 | 0.34147705E+02 | 0.52530615E+03 |
| A X B INTERACTION | 3 | 0.4122?99?E+00 | 0.13745332E+00 | 0.21141?44E+01 |
| ERROR | 32 | 0.20801?0?E+01 | 0.650?5337E-01 | |
| TOTAL | 39 | 0.1054?45?E+03 | | |

TABLE 8

TWO FACTOR ANALYSIS OF VARIANCE
FOR
PEEL DATA, FACTOR A = MAPO SOURCE (CONTROL AND SPECIAL), FACTOR B = TEST PERIOD

| SOURCE | DF | SS | MS | F |
|---|---|---|---|---|
| FACTOR A | 1 | 0.7239795BE-02 | 0.7239795BE-02 | 0.38226741E+01 |
| FACTOR B | 3 | 0.95953271E+00 | 0.31984424E+00 | 0.16888928E+03 |
| A X B INTERACTION | 3 | 0.13248791E-01 | 0.44162637E-02 | 0.23319464E+01 |
| ERROR | 40 | 0.75752380E-01 | 0.1893C095E-02 | |
| TOTAL | 47 | 0.10557737E+01 | | |

TABLE 9

SIGNIFICANCES AT THE FIVE PERCENT SIGNIFICANCE LEVEL
PERTAINING TO ANALYSIS OF VARIANCE TABLES 6, 7, and 8

| Test | MAPO Source (A) | Test Period (B) | A x B Interaction |
|---|---|---|---|
| Disc | NS | S | S |
| Cup | S | S | NS |
| Peel | NS | S | NS |

NS = Not Significant
S = Significant

TABLE 10

FAILURE MODE FOR CUP SPECIMENS 1978 DATA

| CONTROL % | | | SPECIAL % | | |
|---|---|---|---|---|---|
| Specimen Number | Adhesive Propellant to Liner | Cohesive in Propellant | Specimen Number | Adhesive Propellant to Liner | Cohesive in Propellant |
| 79 | 95 | 5 | 259 | 40 | 60 |
| 80 | 40 | 60 | 260 | 50 | 50 |
| 81 | 95 | 5 | 261 | 25 | 75 |
| 82 | 30 | 70 | 262 | 30 | 70 |
| 83 | 5 | 95 | 263 | 50 | 50 |
| 84 | 100 | 0 | 264 | 50 | 50 |
| Average | 61 | 39 | | 41 | 59 |

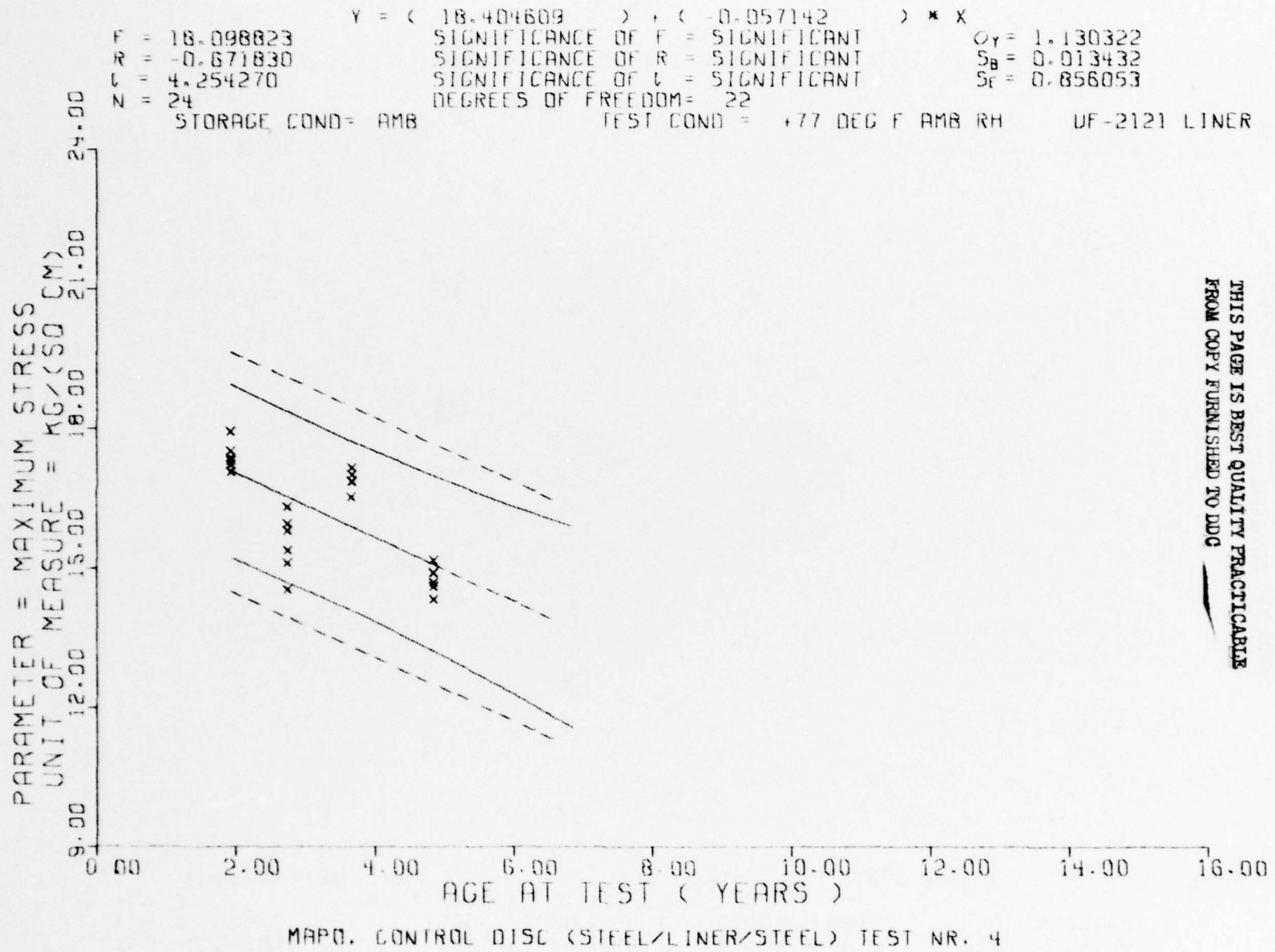







Figure 1

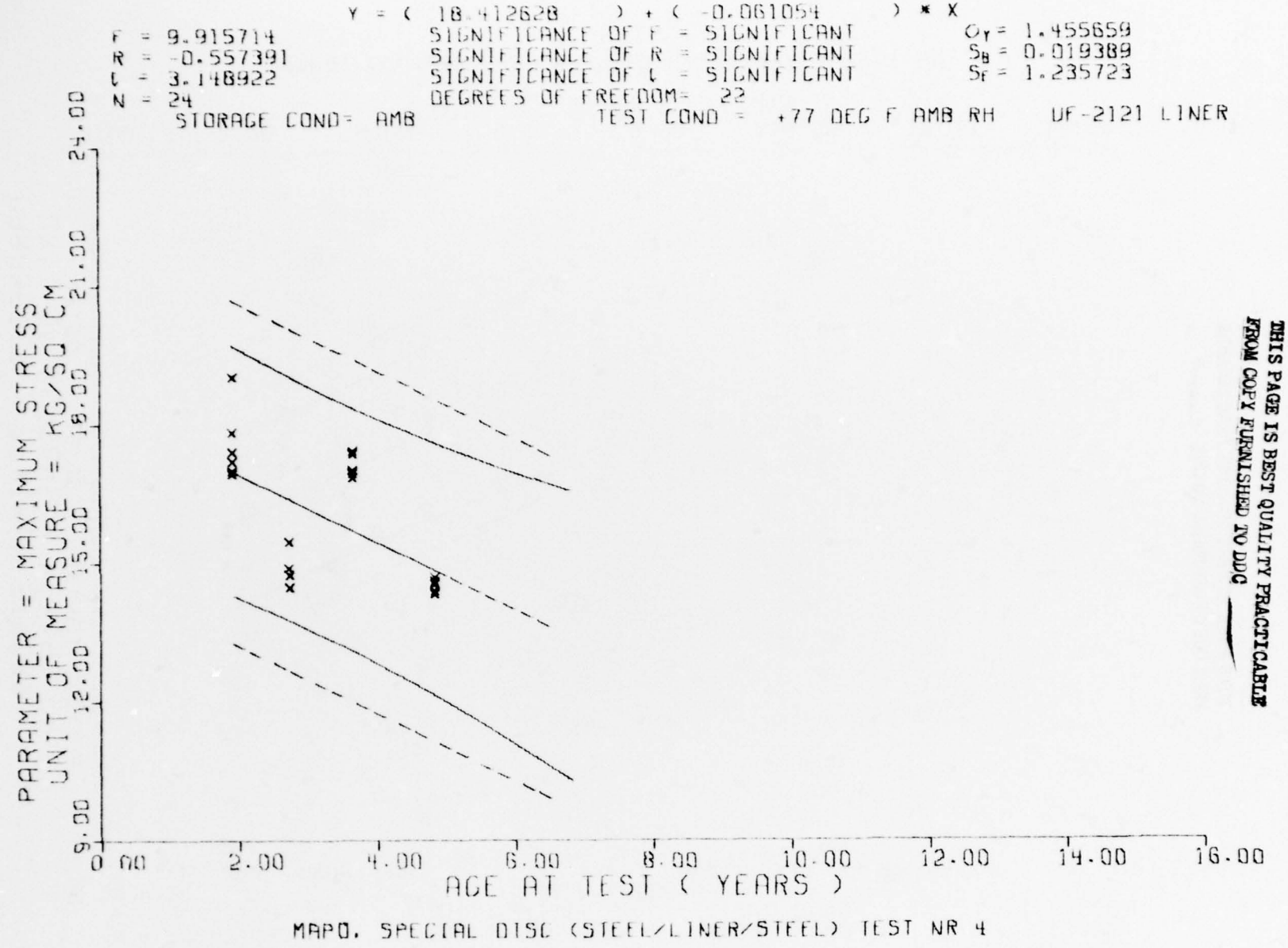



Figure 2

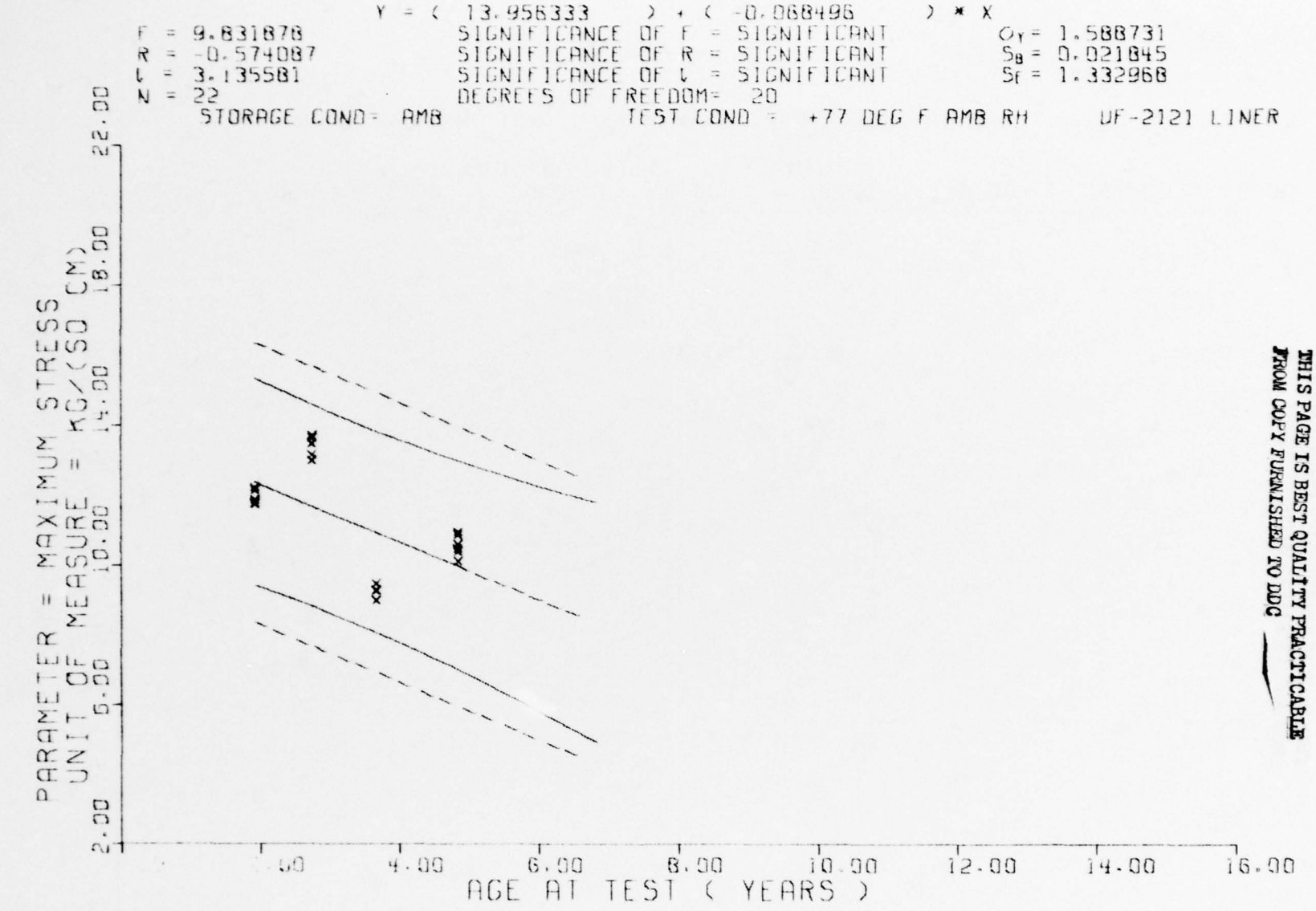



Figure 3

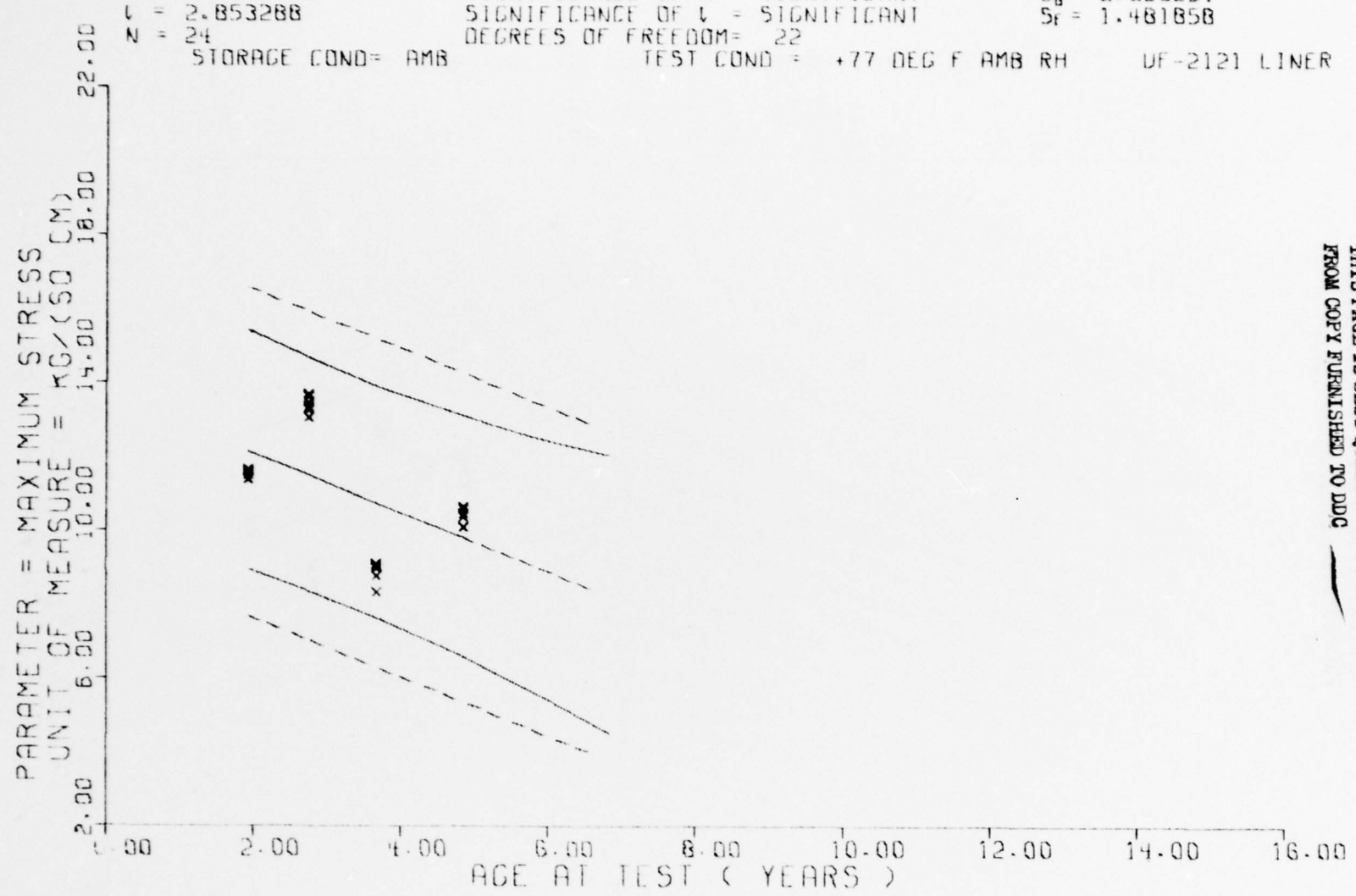
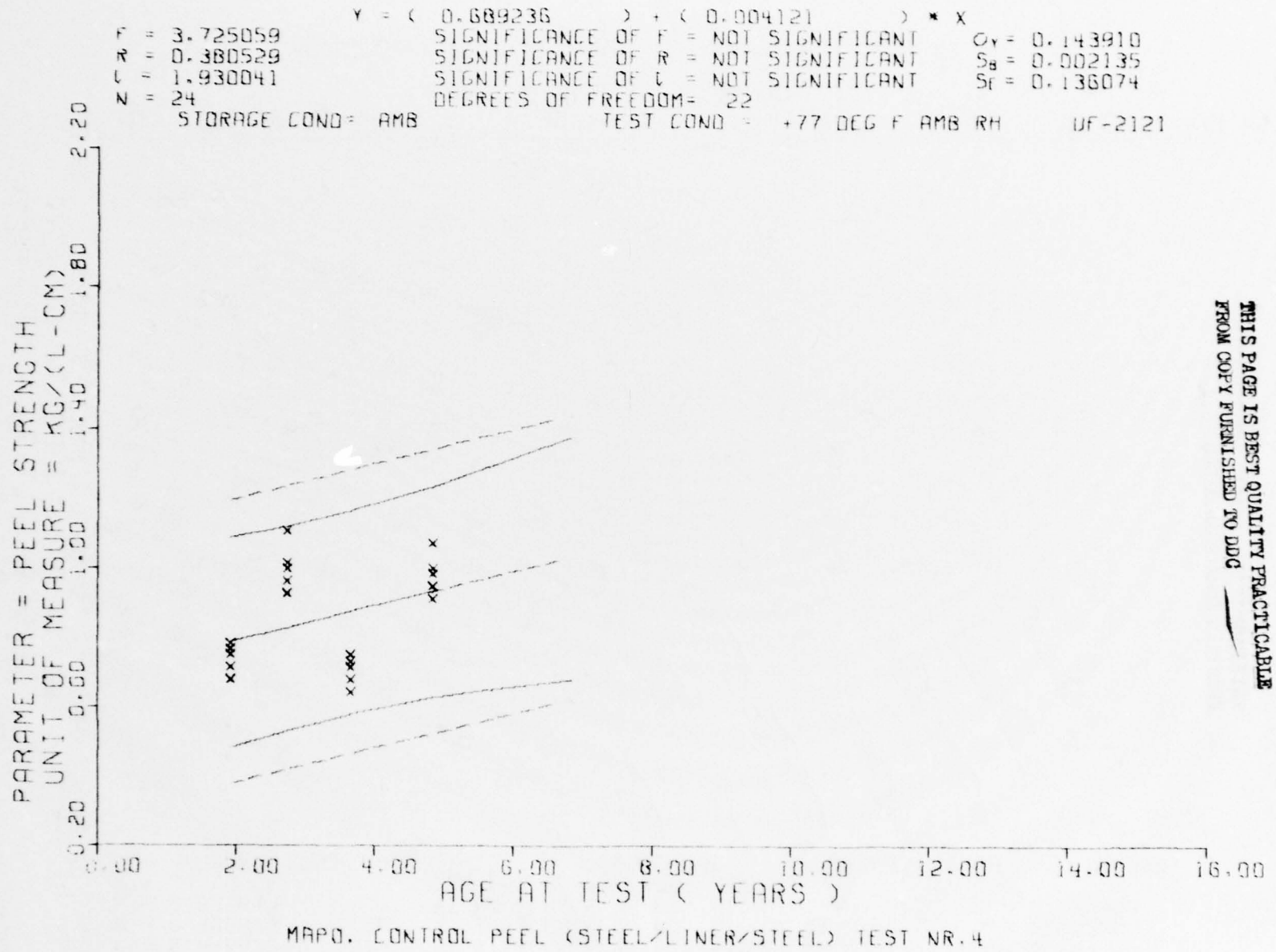







Figure 4

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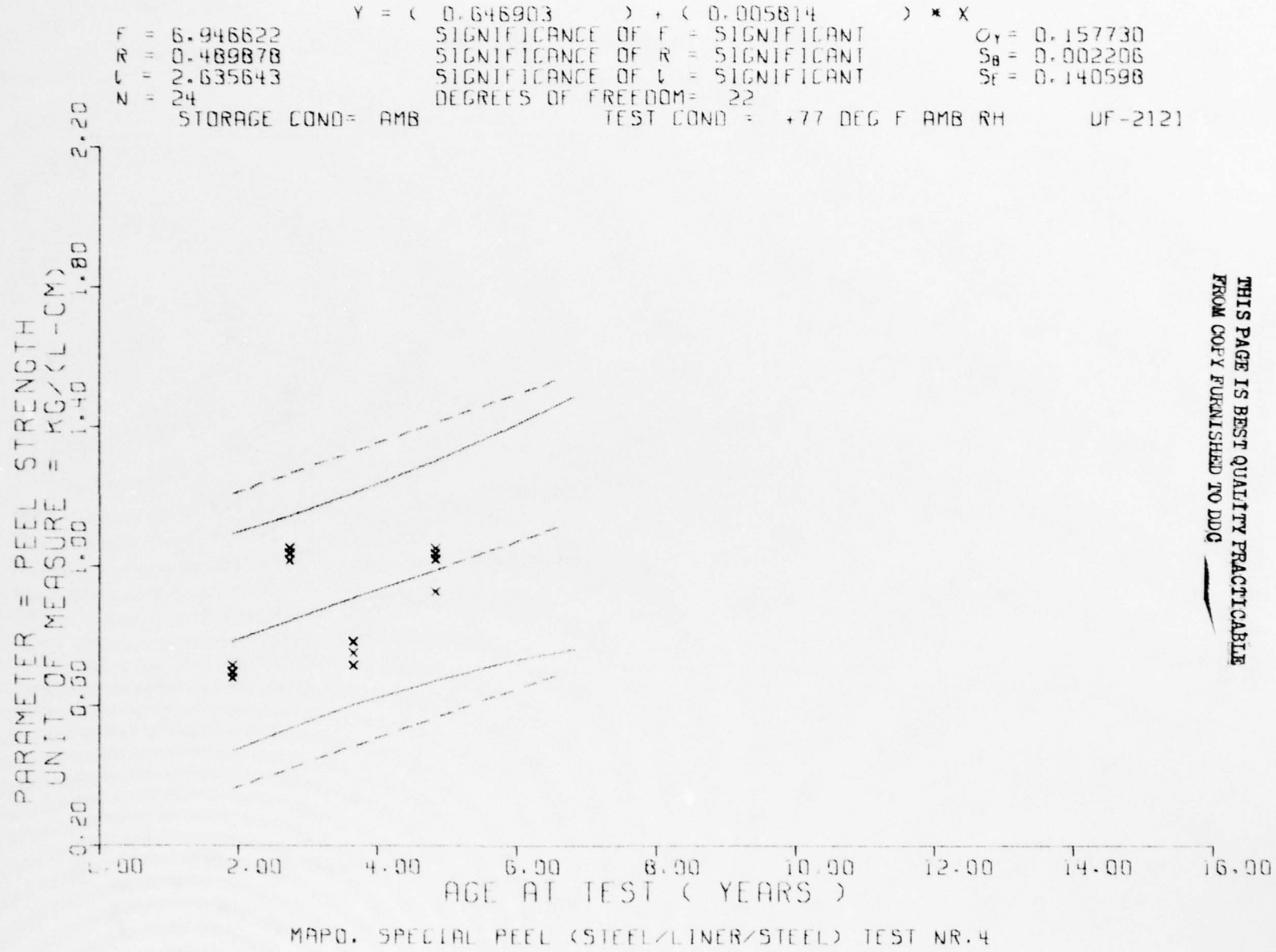


Figure 6

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UF-2121 Liner

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Thiokol Chemical Corporation/Wasatch Division used MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which were used as the control material in the qualification testing. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

This report includes the test results for the first, second, third and fourth

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time testing of the control and special specimens at Ogden ALC.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 Rev A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.